

Standard Operating Procedure for the Determination of Phosphorous and Potassium in Fertilizer by ICP

1.0 Scope and Application

This method is a screening procedure that can be used to determine phosphorous and potassium in fertilizers, using the extract prepared for the direct extraction method 993.31. The same method can be found on SOP number II-2-13.

2.0 Summary

A portion of the direct extraction is put into autosampler tubes and analyzed by ICP. The ICP is calibrated with standards that contain both potassium and phosphorous stock standard solution diluted with the same amount of Ammonium Citrate-EDTA extracting solution as is present in the sample extracts.

3.0 Definitions

- 3.1** The definitions and purposes below are specific to this method, but have been conformed to common usage as much as possible.
- 3.2** This method uses grams (g), milligrams(mg), liters(L), and milliliters(ml). It also requires the use of the acronyms ICP and EDTA.
- 3.3** EDTA - Ethylenediamine Tetraacetate (Disodium)
ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy

4.0 Interferences

- 4.1** A potential problem with the ICP is that the detector can be saturated by the high levels of Potassium and/or Phosphorous. A recommended procedure, if this problem is seen, is to dilute the sample by a 1 to 2 ratio. At this time, there are no other interferences that are seen in this method.

5.0 Safety

- 5.1** There are no special requirements outside of Good Laboratory Practices and common sense. Gloves, safety glasses, and lab coat shall be worn when the use of reagents is required.

6.0 Equipment and Supplies

- 6.1** Inductively Coupled Plasma Emission Spectrometer.
- 6.2** Autosampler tubes.
- 6.3** 100 ml volumetric flasks.
- 6.4** 200 ml volumetric flasks.
- 6.5** Various volumetric pipettes.
- 6.6** Filter paper. (Nonquantitative is suitable.)
- 6.7** 250 ml volumetric wide mouth flasks.
- 6.8** Stoppers.
- 6.9** Heated shaker bath.

7.0 Reagents and Standards

- 7.1** Ammonium Citrate - 100% pure compound.
- 7.2** Ethylenediamine Tetraacetate - 95%.
- 7.3** Ammonium Citrate-EDTA solution: Dissolve 25 g disodium EDTA and 50 g dibasic ammonium citrate in 1.5 L H₂O. Nearly neutralize by adding 30 ml of (1+1) NH₄OH solution and then adjust the pH to 7.00 with the (1+1) NH₄OH solution. Dilute to 2 L with H₂O. I use this formula in 5 times as much.
- 7.4** Stock standards are obtained from the Plasmachem Corporation. The concentration is 10,000 micrograms per ml.
- 7.5** Calibration standards are prepared by measuring the proper amount of stock standard into a 100 ml volumetric flask, adding 40 mL of ammonium citrate-EDTA solution, and diluting to volume with deionized water. Concentrations of phosphorus and potassium in the high and low calibration standards should bracket the concentrations expected in the sample extracts. Three calibration standards are typically prepared. The normal high standard is 1145.60 ppm phosphorous as P₂O₅ and 602.3 ppm potassium as K₂O. The normal mid-level standard is 687.3 ppm phosphorus as P₂O₅ and 301.15 ppm potassium as K₂ O. The normal low

standard 114.55 ppm phosphorous as P_2O_5 and 120.4 ppm potassium as K_2O .

- 7.6** The low-level calibration standard is made by measuring 0.5 ml of phosphorous stock standard and 1.0 ml of potassium stock standard into a 100 ml volumetric flask. The mid-level calibration standard is made by measuring 3.0 ml of phosphorous stock standard and 2.5 ml of potassium stock standard into a 100 ml volumetric flask. The high-level calibration standard is made by measuring 5.0 ml of phosphorous stock standard and 5.0 ml of potassium stock standard into a 100 ml volumetric flask. To each volumetric flask, add 40 ml of ammonium citrate-EDTA and fill to the line with deionized water.

8.0 Sample Collection, Preservation, and Storage

- 8.1** Samples are collected by inspectors from the Department of Agriculture. The samples are collected in bags and brought to the lab. A portion of each sample is ground before analysis. The samples are stored on shelves in the grinding room.
- 8.2** Samples that pass all analytical tests are retained for 3 months. If analysis shows a deficiency for one or more components in a sample, the sample must be kept for 6 months.

9.0 Quality Control

- 9.1** The pH of the ammonium citrate-EDTA should be 7.00. It should be checked before each use and adjusted appropriately.
- 9.2** The shaker bath should be kept in the range of 63 to 67 degrees C.
- 9.3** Analyze a known-value check sample and a duplicate sample for every ten samples. Check samples are prepared exactly the same as a regular sample. The results for the check sample must fall within three standard deviations of the grand average.

10.0 Calibration and Standardization

- 10.1** The ICP has a method by the name of "toddkp." This method has been designed and formatted for this method **only**.
- 10.2** The calibration system is preset. The slope is the best way to tell if the calibration is correct. If the calibration is off, remake the standards and try again.

- 10.3** The calibration data storage is printed out in hard copy and also stored in the computers data bank.

11.0 Procedure

- 11.1** Weigh 0.5 g sample into a 250 wide-mouth volumetric flask. (Record the sample weight to the nearest 0.0001g.)
- 11.2** Fill the shaker bath with distilled water to a minimum of ½ inch above the top of the bottle clamps. Switch on the shaker bath heater.
- 11.3** Transfer 100 ml of the ammonium citrate-EDTA solution into the 250 ml volumetric flask containing the sample and place in the shaker bath. Shake samples for 1 hour at $65 \pm 2^{\circ} \text{C}$ and at 200 rpm.
- 11.4** Remove flasks from shaker bath and let cool. Bring to volume with deionized water and mix thoroughly.
- 11.5** Allow the sample solution to stand for at least two hours or preferably overnight.
- 11.6** Filter the sample if necessary.
- 11.7** Place a portion into a labeled autosampler tube.
- 11.8** The weights should then be entered into the ICP along with the values for the standards used for calibration.

12.0 Data Analysis, Calculations, and Reporting Results

12.1 Calculations

All the calculations can be done by the ICP. The results are given in weight percent, which are converted from parts per million. The general formula used is

$$X\% = \frac{(0.25L)(\text{PPM of sample})(100)(\text{dilution factor})}{(\text{milligrams of sample})}$$

13.0 Method Performance

This method was developed in our own laboratory. The results coming from this method were verified by using check samples from the Association of Florida Phosphate Chemists and also using Magruder Fertilizer Check samples.

14.0 Pollution Prevention

No applicable procedure is available at this time.

15.0 Waste Management

For information on waste management consult The Waste Management Manual for Laboratory Personnel and Less is Better: Laboratory Chemical Management for Waste Reduction, both available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street N.W., Washington, D.C. 20036.

16.0 References

Official Methods of Analysis (1984) 14th Ed, AOAC, Washington, D.C.